State of California
Air Resources Board

Method 11

Determination of Hydrogen Sulfide Content of Fuel Gas Streams in Petroleum Refineries

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Amended: _____

Note: this document consists of the text of the proposed amendment to Method 11. Proposed deletions are noted by graphic screen and proposed additions are noted by underline.

Method 11 - Determination of Hydrogen Sulfide Content of Fuel Gas Streams in Petroleum Refineries

1. PRINCIPLE AND APPLICABILITY

1.1 Principle. Hydrogen sulfide (H_2S) is collected from a source in a series of midget impingers and absorbed in pH 3.0 cadmium sulfate ($CdSO_4$) solution to form cadmium sulfide (CdS). The latter compound is then measured iodometrically. An impinger containing hydrogen peroxide (H_2O_2) is included to remove SO_2 as an interfering species.

1.2 Applicability. This method is applicable for the determination of the hydrogen sulfideH_2S content of fuel gas streams at petroleum refineries.

Any modification of this method beyond those expressly permitted shall be considered a major modification subject to the approval of the Executive Officer. The term Executive Officer as used in this document shall mean the Executive Officer of the Air Resources Board (ARB), or his or her authorized representative.

2. RANGE AND SENSITIVITY

The lower limit of detection is approximately 8 mg/m^3 (6 ppm). The maximum of the range is 740 mg/m^3 (520 ppm).

3. INTERFERENCES

3.1 Any compound that reduces iodine (I_2) or oxidizes iodide ion will interfere in this procedure, provided it is collected in the $CdSO_4$ impingers. Sulfur dioxide in concentrations of up to 2,600 mg/m^3 is eliminated by the H_2O_2 solution. Thiols precipitate with H_2S. In the absence of H_2S, only co-traces of thiols are collected. When methane- and ethane-thiols at a total level of 300 mg/m^3 are present in addition to H_2S, the results vary from 2 percent low at an H_2S concentration of 400 mg/m^3 to 14 percent high at an H_2S concentration of 100 mg/m^3. Carbon oxysulfide at a concentration of 20 percent does not interfere. Certain carbonyl-containing compounds react with iodine and produce recurring end points. However, acetaldehyde and acetone at concentrations of 1 and 3 percent, respectively, do not interfere.

3.2 Entrained H_2O_2 produces a negative interference equivalent to 100 percent of that of an equimolar quantity of hydrogen sulfideH_2S. Avoid the ejection of H_2O_2 into the $CdSO_4$ impingers.

4. PRECISION AND ACCURACY

Collaborative testing has shown the within-laboratory coefficient of variation to be 2.2 percent and the overall coefficient of variation to be 5 percent. The method bias was shown to be -4.8 percent when only H_2S was present. In the presence of the interferences cited in Section 3, the bias was positive at low H_2S concentration and negative at higher concentrations. At

230 mg H_2S/m^3, the level of the compliance standard, the bias was +2.7 percent. Thiols had no effect on the precision.

5. APPARATUS

Note:(this note formerly appeared as a footnote) Mention of trade names or specific products does not constitute endorsement by the Air Resources Board.

5.1 Sampling Apparatus.

5.1.1 Sampling Line. Six to 7 mm Teflon tubingTeflon tubing, 6- to 7-mm (1/4-in.) ID, to connect the sampling train to the sampling valve.

5.1.2 Impingers. Five midget impingers, each with 30-ml capacity. The internal diameter of the impinger tip must be 1 mm ± 0.05 mm. The impinger tip must be positioned 4 to 6 mm from the bottom of the impinger.

5.1.3 Tubing. Glass or Teflon connecting tubing for the impingers.

5.1.4 Ice Bath Container. To maintain absorbing solution at a low temperature.

5.1.5 Drying Tube. Tube packed with 6- to 16-mesh indicating-type silica gel, or equivalent, to dry the gas sample and protect the meter and pump. If the silica gel has been used previously, dry at 175 C (350 F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to approval of the Control Agency's Authorized RepresentativeExecutiveOfficer.

Note: Do not use more than 30 g of silica gel. Silica gel adsorbs gases such as propane from the fuel gas stream, and use of excessive amounts of silica gel could result in errors in the determination of sample volume.

5.1.6 Sampling Valve. Needle valve, or equivalent, to adjust gas flow rate. Stainless steel or other corrosion-resistant material.

5.1.7 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, calibrated at the selected flow rate (about 1.0 liter/min) and conditions actually encountered during sampling. The meter shall be equipped with a temperature gauge (dial thermometer or equivalent) capable of measuring temperature to within 3 C (5.4 F). The gas meter should have a petcock, or equivalent, on the outlet connector which can be closed during the leak check. Gas volume for one revolution of the meter must not be more than 10 liters.

5.1.8 Flow Meter. Rotameter, or equivalent, to measure flow rates in the range from 0.5 to 2 liters/min (1 to 4 cfh).

5.1.9 Graduated Cylinder. 25-ml size.

5.1.10 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby National Weather Service station, in which case, the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in Hg) per 30 m (100 ft) elevation increase or vice-versa for elevation decrease.

5.1.11 U-tube Manometer. 0-30 cm0- to 30-cm water column., Ffor leak-check procedure.

5.1.12 Rubber Squeeze Bulb. To pressurize train for leak-check.

5.1.13 Tee, Pinchclamp, and Connecting Tubing. For leak-check.

5.1.14 Pump. Diaphragm pump, or equivalent. Insert a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter. The pump is used for the air purge at the end of the sample run; the pump is not ordinarily used during sampling, because fuel gas streams are usually sufficiently pressurized to force sample gas through the train at the required flow rate. The pump need not be leak-free unless it is used fromfor sampling.

5.1.15 Needle Valve or Critical Orifice. To set air purge flow to 1 liter/min.

5.1.16 Tube Packed with Active Carbon. To filter air during purge.

5.1.17 Volumetric Flask. One 1000-ml.

5.1.18 Volumetric Pipette. One 15-ml.

5.1.19 Pressure-Reduction Regulator. Depending on the sampling stream pressure, a pressure-reduction regulator may be needed to reduce the pressure of the gas stream entering the Teflon sample line to a safe level.

5.1.20 Cold Trap. If condensed water or amine is present in the sample stream, a corrosion-resistant cold trap shall be used immediately after the sample tap. The trap shall not be operated below 0 C (32 F) to avoid condensation of C3 or C4 hydrocarbons.

5.2 Sample Recovery.

5.2.1 Sample Container. Iodine flask, glass-stoppered, 500-ml size.

5.2.2 Volumetric Pipette. One 50-ml.

5.2.3 Graduated Cylinders. One each 25- and 250-ml.

5.2.4 Erlenmeyer Flasks. 125-ml.

5.2.5 Wash Bottle.

5.2.6 Volumetric Flasks. Three l,000-ml.

5.3 Analysis.

5.3.1 Flask. 500-ml Glass-stoppered iodine flask, 500-ml.

5.3.2 Burette. 50-ml.

5.3.3 Erlenmeyer Flask. 125-ml.

5.3.4 Pipettes, Volumetric Volumetric Pipettes. One 25 ml; two each 50 and 100 ml. One 25-ml; two each 50- and 100-ml.

5.3.5 Volumetric Flasks. One l,000 ml; two 500 ml. One l,000-ml; two 500-ml.

5.3.6 Graduated Cylinders. One each 10 and 100 ml. One each 10- and 100-ml.

6. REAGENTS

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Otherwise, use best available grade.

6.1 Sampling.

6.1.1 Cadmium Sulfate $CdSO_4$ Absorbing Solution. Dissolve 41 g of $3CdSO_4 \cdot 8H_2O$ and 15 ml of 0.1 M sulfuric acid in a 1-liter volumetric flask that contains approximately 3/4 liter of deionized, distilled water. Dilute to volume with deionized, distilled water. Mix thoroughly. The pH should be 3±0.1. Add 10 drops of Dow-Corning Antifoam B. Shake well before use. If Antifoam B is not used, the alternative acidified iodine extraction procedure (Section 7.2.2) must be used.

6.1.2 Hydrogen Peroxide H_2O_2, 3 Percent. Dilute 30 percent H_2O_2 to 3 percent as needed. Prepare fresh daily.

6.1.3 Water. Deionized distilled to conform to ASTM Specification D 1193-72, Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

6.2 Sample Recovery.

6.2.1 Water. Same as Section 6.1.3.

6.2.12 Hydrochloric Acid (HCl) Solution (HCl), 3 M. Add 240 ml of concentrated HCl (specific gravity 1.19) to 500 ml of deionized, distilled water in a 1-liter volumetric flask. Dilute to 1 liter with water. Mix thoroughly.

6.2.23 Iodine Solution, 0.1 N. Dissolve 24 g of potassium iodide (KI) in 30 ml of water. Add 12.7 g of resublimed iodine (I_2) to the potassium iodideKI solution. Shake the mixture until the iodineI_2 is completely dissolved. If possible, let the solution stand overnight in the dark. Slowly dilute the solution to 1 liter with deionized, distilled water, with swirling. Filter the solution if it is cloudy. Store solution in a brown-glass reagent bottle.

6.2.34 Standard IodineI_2 Solution, 0.01 N. Pipette 100.0 ml of the 0.1 N iodine solution into a l-liter volumetric flask, and dilute to volume with deionized, distilled water. Standardize daily as in Section 8.1.1. This solution must be protected from light. Reagent bottles and flasks must be kept tightly stoppered.

6.3 Analysis.

6.3.1 Water. Same as in Section 6.1.3.

6.3.12 Standard Sodium Thiosulfate Solution, 0.1 N. Dissolve 24.8 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) or 15.8 g of anhydrous sodium thiosulfate (Na2S203) in 1 liter of water, and add 0.01 g of anhydrous sodium carbonate (Na_2CO_3) and 0.4 ml of chloroform ($CHCl_3$) to stabilize. Mix thoroughly by shaking or by aerating with nitrogen for approximately 15 minutes, and store in a glass-stoppered, reagent bottle. Standardize as in Section 8.1.2.

6.3.23 Standard Sodium Thiosulfate Solution, 0.01 N. Pipette 50.0 ml of the standard 0.1 N thiosulfate$Na_2S_2O_3$ solution into a volumetric flask, and dilute to 500 ml with water.

Note: A 0.01 N phenylarsine oxide (C_6H_5AsO) solution may be prepared instead of 0.01 N thiosulfate$Na_2S_2O_3$ (see Section 6.3.34).]

6.3.34 Phenylarsine Oxide Solution, Standard 0.01 N. Dissolve 1.80 g of phenylarsine oxide(C6H5As0) in 150 ml of 0.3 N sodium hydroxide. After settling, decant 140 ml of this solution into 800 ml of distilled water. Bring the solution to pH 6-7 with 6 N hydrochloric acidHCl, and dilute to 1 liter with water. Standardize as in Section 8.1.3.

6.3.45 Starch Indicator Solution. Suspend 10 g of soluble starch in 100 ml of deionized, distilled water, and add 15 g of potassium hydroxide (KOH) pellets. Stir until dissolved, dilute with 900 ml of deionized, distilled water, and let stand for 1 hour. Neutralize the alkali with concentrated hydrochloric acidHCl, using an indicator paper similar to Alkacid test ribbon, then add 2 ml of glacial acetic acid as a preservative.

Note: Test starch indicator solution for decomposition by titrating with 0.01 N iodine I_2 solution, 4 ml of starch solution in 200 ml of distilled water that contains 1 g of potassium iodide KI. If more than 4 drops of the 0.01 N iodine I_2 solution are required to obtain the blue color, a fresh solution must be prepared.)

7. PROCEDURE

7.1 Sampling.

7.1.1 Assemble the sampling train as shown in Figure 11-1, connecting the five midget impingers in series. Place 15 ml of 3 percent hydrogen peroxide H_2O_2 solution in the first impinger. Leave the second impinger empty. Place 15 ml of the cadmium sulfate $CdSO_4$ solution in the third, fourth, and fifth impingers. Place the impinger assembly in an ice bath container, and place crushed ice around the impingers. Add more ice during the run, if needed.

7.1.2 Connect the rubber bulb and manometer to the first impinger, as shown in Figure 11-1. Close the petcock on the dry gas meter outlet. Pressurize the train to 25 cm 25-cm water with the bulb, and close off the tubing connected to the rubber bulb. The train must hold a 25-cm water pressure with not more than a 1-cm drop in pressure in a 1-minute interval. Stopcock grease is acceptable for sealing ground glass joints.

Note: This leak-check procedure is to be conducted at the beginning and the end of the sample run optional at the beginning of the sample run, but is mandatory at the conclusion. Note also that if the pump is used for sampling, it is recommended (but not required) that the pump be leak-checked separately, using a method consistent with the leak-check procedure for diaphragm pumps outlined in Section 4.1.2 of Method 6.]

7.1.3 Purge the connecting line between the sampling valve and the first impinger, by disconnecting the line from the first impinger, opening the sampling valve, and allowing process gas to flow through the line for a minute or two. Then, close the sampling valve, and reconnect the line to the impinger train. Open the petcock on the dry gas meter outlet. Record the initial dry gas meter reading.

7.1.4 Open the sampling valve, and then adjust the valve to obtain a rate of approximately 1 liter/min. Maintain a constant (±10 percent) flow rate during the test. Record the meter temperature.

7.1.5 Sample for at least 10 minutes. At the end of the sampling time, close the sampling valve, and record the final volume and temperature readings. Conduct a leak-check as described in Section 7.1.2 above.

7.1.6 Disconnect the impinger train from the sampling line. Connect the charcoal tube and the pump, as shown in Figure 11-1. Purge the train (at a rate of 1 liter/min) with clean ambient air for 15 minutes to ensure that all H_2S is removed

from the hydrogen peroxideH_2O_2. For sample recovery, cap the open ends, and remove the impinger train to a clean area that is away from sources of heat. The area should be well lighted, but not exposed to direct sunlight.

7.2 Sample Recovery.

7.2.1 Discard the contents of the hydrogen peroxideH_2O_2. impinger. Carefully rinse with water the contents of the third, fourth, and fifth impingers into a 500-ml iodine flask.

Note: The impingers normally have only a thin film of cadmium sulfideCdS remaining after a water rinse. If Antifoam B was not used or if significant quantities of yellow cadmium sulfideCdS remain in the impingers, the alternate recovery procedure described below must be used.

7.2.2 Pipette exactly 50 ml of 0.01 N iodineI_2 solution into a 125-ml Erlenmeyer flask. Add 10 ml of 3 M HCl to the solution. Quantitatively rinse the acidified iodineI_2 into the iodine flask. Stopper the flask immediately, and shake briefly.

(Alternate). Extract the remaining cadmium sulfideCdS from the third, fourth, and fifth impingers using the acidified iodineI_2 solution. Immediately after pouring the acidified iodineI_2 into an impinger, stopper it and shake for a few moments, then transfer the liquid to the iodine flask. Do not transfer any rinse portion from one impinger to another; transfer it directly to the iodine flask. Once the acidified iodineI_2 solution has been poured into any glassware containing CdS, the container must be tightly stoppered at all times except when adding more solution, and this must be done as quickly and carefully as possible. After adding any acidified I_2 solution to the iodine flask, allow a few minutes for absorption of the H_2S before adding any further rinses. Repeat the iodineI_2 extraction until all cadmium sulfideCdS is removed from the impingers. Extract that part of the connecting glassware that contains visible cadmium sulfideCdS.

Quantitatively rinse all the iodineI_2 from the impingers, connectors, and the beaker into the iodine flask using water. Stopper the flask and shake briefly.

7.2.3 Allow the iodine flask to stand about 30 minutes in the dark for absorption of the H_2S into the iodineI_2, then complete the titration analysis as in Section 7.3. Note: Caution! Iodine evaporates from acidified iodineI_2 solutions. Samples to which acidified iodineI_2 have been added may not be stored, but must be analyzed in the time schedule stated above.

7.2.4 Prepare a blank by adding 45 ml of cadmium sulfate$CdSO_4$ absorbing solution to an iodine flask. Pipette exactly 50 ml of 0.01 N iodineI_2 solution into a 125-ml Erlenmeyer flask. Add 10 ml of 3 M HCl. Follow the same impinger extracting and quantitative analysis procedures carried out in sample analysis. Stopper the flask, shake briefly, let stand 30 minutes in the dark, and titrate with the samples.

Note: The blank must be handled by exactly the same procedure as that used for the samples.

7.3 Analysis.

Note: Titration analyses should be conducted at the sample-cleanup area in order to prevent loss of iodineI_2 from the sample. Titration should never be made in direct sunlight.

7.3.1 Using 0.01 N sodium thiosulfate$Na_2S_2O_3$ solution (or 0.01 N phenylarsine oxideC_6H_5AsO, if applicable), rapidly titrate each sample in an iodine flask using gentle mixing, until solution is light yellow. Add 4 ml of starch indicator solution, and continue titrating slowly until the blue color just disappears. Record V_{TT}, the volume of sodium thiosulfate$Na_2S_2O_3$ solution used, or V_{AT}, the volume of phenylarsine oxideC_6H_5AsO solution used, (ml)in ml.

7.3.2 Titrate the blanks in the same manner as the samples. Run blanks each day until replicate values agree within 0.05 ml. Average the replicate titration values which agree within 0.05 ml.

8. CALIBRATION AND STANDARDS

8.1 Standardizations.

8.1.1 Standardize the 0.01 N iodineI_2 solution daily as follows: Pipette 25 ml of the iodineI_2 solution into a 125-ml Erlenmeyer flask. Add 2 ml of 3 M HCl. Titrate rapidly with standard 0.01 N thiosulfate$Na_2S_2O_3$ solution or with 0.01 N phenylarsine oxideC_6H_5AsO until the solution is light yellow, using gentle mixing. Add four drops of starch indicator solution, and continue titrating slowly until the blue color just disappears. Record V_T, the volume of thiosulfate$Na_2S_2O_3$ solution used, or V_{AS}, the volume of phenylarsine oxideC_6H_5AsO solution used, (ml)in ml. Repeat until replicate values agree within 0.05 ml. Average the replicate titration values which agree within 0.05 ml, and calculate the exact normality of the iodineI_2 solution using equation 9.3Equation 11-3. Repeat the standardization daily.

8.1.2 Standardize the 0.1 N thiosulfate$Na_2S_2O_3$ solution as follows: Oven-dry potassium dichromate ($K_2Cr_20_7$) at 180 to 200° C (360 to 390° F). Weigh to the nearest milligram, 2 g of potassiumthe dichromate. Transfer the dichromate to a 500-ml volumetric flask, dissolve in deionized, distilled water and dilute to exactly 500 ml. In a 500 ml iodine flask, dissolve approximately 3 g of potassium iodide(KI) in 45 ml of deionized, distilled water, then add 10 ml of 3 M hydrochloric acidHCl solution. Pipette 50 ml of the dichromate solution into this mixture. Gently swirl the solution once, and allow it to stand in the dark for 5 minutes. Dilute the solution with 100 to 200 ml of deionized, distilled water, washing down the sides of the flask with part of the water. Titrate with 0.1 N thiosulfate$Na_2S_2O_3$ until the solution is light yellow. Add 4 ml of starch indicator and continue titrating slowly to a green end point. Record V_S, the volume of thiosulfate$Na_2S_2O_3$ solution used, (ml)in ml.

Repeat until replicate analyses agree within 0.05 ml. Calculate the normality using equation 9.1Equation 11-1. Repeat the standardization each week, or after each test series, whichever time is shorter.

8.1.3 Standardize the 0.01 N phenylarsine oxideC_6H_5AsO (if applicable) as follows: Oven-dry potassium dichromate ($K_2Cr_20_7$) at 180 to 200° C (360 to 390° F). Weigh to the nearest milligram, 2 g of the $K_2Cr_20_7$ dichromate; transfer the dichromate to a 500-ml volumetric flask, dissolve in deionized, distilled water, and dilute to exactly 500 ml. In a 500-ml iodine flask, dissolve approximately 0.3 g of potassium iodide(KI) in 45 ml of deionized, distilled water; add 10 ml of 3 M hydrochloric acidHCl. Pipette 5 ml of the $K_2Cr_20_7$ dichromate solution into the iodine flask. Gently swirl the contents of the flask once and allow to stand in the dark for 5 minutes. Dilute the solution with 100 to 200 ml of deionized, distilled water, washing down the sides of the flask with part of the water. Titrate with 0.01 N phenylarsine oxideC_6H_5AsO until the solution is light yellow. Add 4 ml of starch indicator, and continue titrating slowly to a green end point. Record V_A, the volume of phenylarsine oxideC_6H_5AsO used, (ml)in ml. Repeat until replicate analyses agree within 0.05 ml. Calculate the normality using equation 9.2Equation 11-2. Repeat the standardization each week or after each test series, whichever time is shorter.

8.2 Sampling Train Calibration. Calibrate the sampling train components as follows:

8.2.1 Dry Gas Meter.

8.2.1.1 Initial Calibration. The dry gas meter shall be calibrated before its initial use in the field. Proceed as follows: First, assemble the following components in series: Drying tube, needle valve, pump, rotameter, and dry gas meter. Then, leak-check the system as follows: Place a vacuum gauge (at least 760 mm Hg) at the inlet to the drying tube, and pull a vacuum of 250 mm (10 in.) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end. Next, calibrate the dry gas meter (at the sampling flow rate specified by the method) as follows: Connect an appropriately sized wet test meter (e.g., 1 liter per revolution) to the inlet of the drying tube. Make three independent calibration runs, using at least five revolutions of the dry gas meter per run. Calculate the calibration factor., Y (wet test meter calibration volume divided by the dry gas meter volume, both volumes adjusted to the same reference temperature and pressure), for each run, and average the results. If any Y value deviates by more than 2 percent from the average, the dry gas meter is unacceptable for use. Otherwise, use the average as the calibration factor for subsequent test runs.

8.2.1.2 Post-Test Calibration Check. After each field test series, conduct a calibration check as in Section 8.2.1.1, above, except for the following variations: (a) the leak-check is not to be conducted, (b) three or more revolutions of the dry gas meter may be used, and (c) only two independent

runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor (determined in Section 8.2.1.1), then the dry gas meter volumes obtained during the test series are acceptable. If the calibration factor deviates by more than 5 percent, recalibrate the dry gas meter as in Section 8.2.1.1, and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

8.2.2 Thermometers. Calibrate against mercury-in-glass thermometers.

8.2.3 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

8.2.4 Barometer. Calibrate against a mercury barometer.

9. CALCULATIONS

Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off results only after the final calculation.

9.1 Normality of the Standard (0.1 N) Thiosulfate Solution.

$$N_S = \frac{2.039\ W}{V_S} \qquad \text{Eq. 11-1}$$

Where:

W = Weight of $K_2Cr_20_7$ used, g.

V_S = Volume of $Na_2S_2O_3$ solution used, ml.

N_S = Normality of standard thiosulfate$Na_2S_2O_3$ solution, g-eq/liter.

2.039 = Conversion factor
= (6. eq I_2/mole $K_2Cr_20_7$)(1,000 ml/liter) /= (294.2 g $K_2Cr_20_7$/mole)(10 aliquot factor)

2.039 = Conversion factor =(6 eq I_2/mole $K_2Cr_20_7$)(1,000 ml/liter) divided by [(294.2 g $K_2Cr_20_7$/mole)(10 aliquot factor)]

9.2 Normality of Standard Phenylarsine Oxide Solution (If Applicable).

$$N_A = \frac{0.2039\ W}{V_A} \qquad \text{Eq. 11-2}$$

Where:

W = Weight of $K_2Cr_20_7$ used, ml.

V_A = Volume of $C_6H_5A_5O$ C_6H_5AsO used, ml.

N_A = Normality of standard phenylarsine oxideC_6H_5AsO solution, g=eq/liter g-eq/liter.

2.039 = Conversion factor
= (6. eq. I_2/mole $K_2Cr_20_7$)(1,000 ml/liter) /= (294.2 g $K_2Cr_20_7$/mole)(10 aliquot factor)

2.039 = Conversion factor =(6 eq I_2/mole $K_2Cr_20_7$)(1,000 ml/liter) divided by [(294.2 g $K_2Cr_20_7$/mole)(10 aliquot factor)]

9.3 Normality of Standard Iodine Solution.

$$N_I = \frac{N_T\ V_T}{V_I} \qquad \text{Eq. 11-3}$$

Where:
N_I = Normality of standard iodineI_2 solution, g-eq/liter.

V_I = Volume of standard iodineI_2 solution used, g.

N_T = Normality of standard (0.01 N) thiosulfate$Na_2S_2O_3$ solution; assumed to be 0.1 N_S. g-eq/liter.

V_T = Volume of thiosulfate$Na_2S_2O_3$ solution used, ml.

Note: If phenylarsine oxideC_6H_5AsO is used instead of thiosulfate$Na_2S_2O_3$, replace N_T and V_T in Equation 11-3 with N_A and V_{AS}, respectively (see Sections 8.1.1 and 8.1.3).

9.4 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C and 760 mm Hg).

$$V_{m(std)} = V_m\ Y\ \frac{T_{std}}{T_m}\frac{P_{bar}}{P_{std}} \qquad \text{Eq. 11-4}$$

Where:

$V_{m(std)}$ = Volume of sample through the dry gas meter, standard liters. gas sample at standard conditions, liters.

V_m = Volume of gas sample through the dry gas meter at meter conditions, liters.

T_{std} = Absolute temperature at standard conditions Standard absolute temperature, 293 K.

T_m = Average dry gas meter temperature, K.

P_{bar} = Barometric pressure at the sampling site, mm Hg.

P_{std} = Absolute pressure at standard conditionsStandard absolute pressure, 760 mm Hg.

Y = Dry gas meter calibration factor.

9.5 Concentration of H_2S. Calculate the concentration of H_2S in the gas stream at standard conditions using the following equation:

Old Equation to be deleted:

$$C_{H2S} = K \frac{[\,(V_{IT}\,N_I - V_{TT}\,N_T)_{sample} - (V_{IT}\,N_I - V_{TT}\,N_T)_{blank}\,]}{V_{m(std)}}$$

New Equation to be inserted:

$$C_{H2S} = 17.04 \times 10^3 \frac{(V_{IT}\,N_I - V_{TT}\,N_T)_{sample} - (V_{IT}\,N_I - V_{TT}\,N_T)_{blank}}{V_{m(std)}} \quad \text{Eq. 11-5}$$

Where(metric units):

C_{H2S} = Concentration of H_2S at standard conditions, mg/dscm.

K = Conversion factor = 17.04

= (34.07 g/mole H_2S)(1,000 liters/m^3)(1,000 mg/g) /= 1,000 ml/liter)(2H_2S eq/mole)

17.04×10^3 = Conversion factor = (34.07 g/mole H_2S)(1,000 liters/m^3)(1,000 mg/g) divided by [(1,000 ml/liter)(2H_2S eq/mole)]

V_{IT} = Volume of standard iodineI_2 solution, 50 ml.

N_I = Normality of standard iodineI_2 solution, g-eq/liter.

V_{TT} = Volume of standard (0.01 N) sodium thiosulfate$Na_2S_2O_3$ solution, ml.

N_T = Normality of standard sodium thiosulfate$Na_2S_2O_3$ solution, g-eq/liter.

$V_{m(std)}$ = Standard dry gas volume, liters.

Note: If phenylarsine oxideC_6H_5AsO is used instead of thiosulfate$Na_2S_2O_3$, replace N_T and V_{TT} in equation 9.5Equation 11-5 with N_A and V_{AT}, respectively (see Sections 7.3.1

and 8.1.3).

10. STABILITY

The absorbing solution is stable for at least 1 month. Sample recovery and analysis should begin within 1 hour of sampling to minimize oxidation of the acidified CdS. Once iodine I_2 has been added to the sample, the remainder of the analysis procedure must be completed according to Sections 7.2.2 through 7.3.2.

BIBLIOGRAPHY

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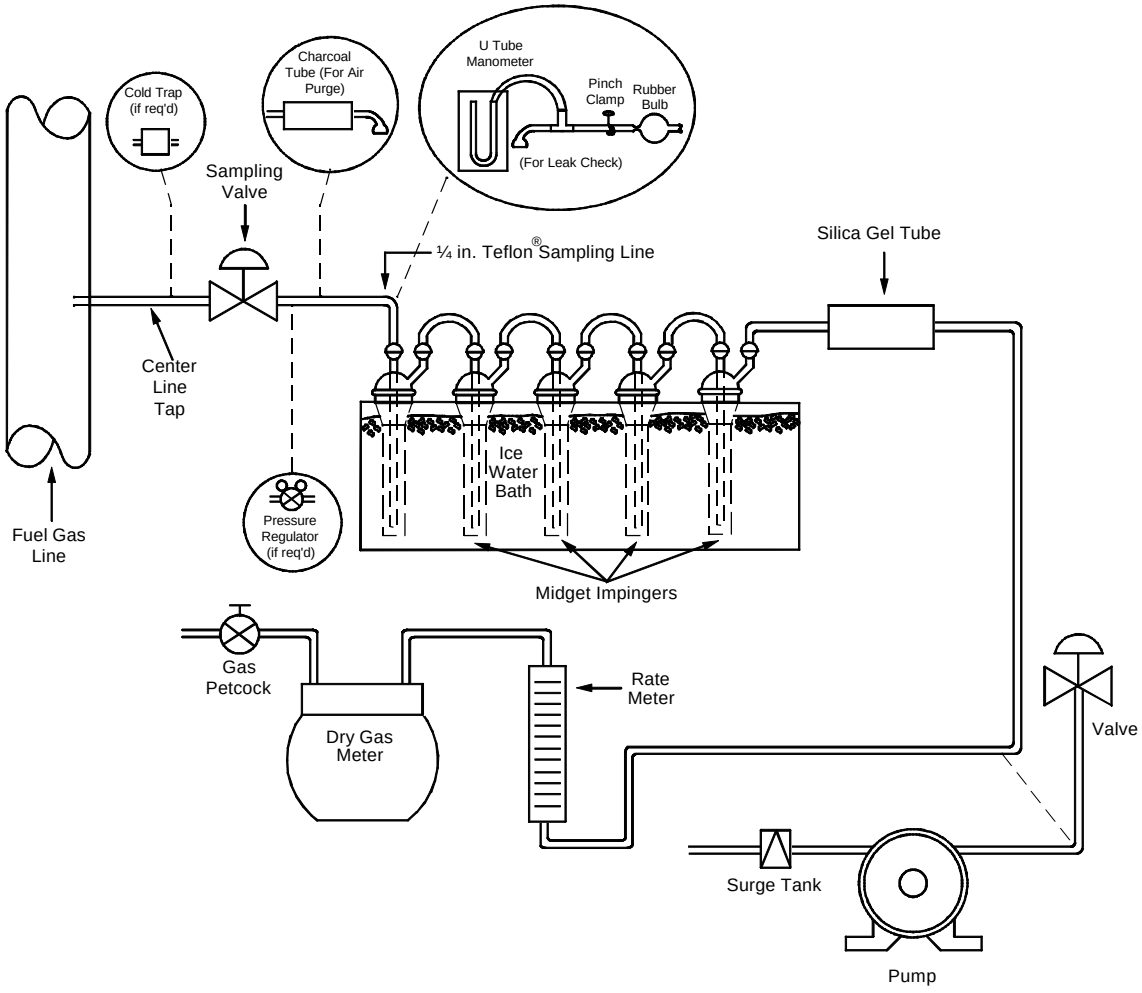

Figure 11-1. H_2S Sampling Train